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Express Mail Label No.: EV 208802493 US
PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant: MATSUMURA et al.

)

S.N.: 09/761,043

)

Examiner: Daniel S. Metzmaier

Filed: January 15, 2001

)

Art Unit: 1712

Confirmation No: 4250

)

For: COLLOIDAL SILICA SLURRY

)

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Marigelle Malapira, Legal Assistant

SUPPLEMENTAL TO
RESPONSE AFTER NON-FINAL OFFICE ACTION

RULE 132 DECLARATION

MS: Box (NON-FEE) Amendment
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Dear Sir:

Applicants responded to the **NON-FINAL** Examiner's Action mailed April 23, 2003 (Paper No. 7), having a shortened statutory period for response which expired on July 23, 2003, and was petitioned to be extended to October 23, 2003, by communication dated October 14, 2003 and mailed by express mail EV 207882668 US.

The undersigned was not aware that Applicants were also sending a Rule 132 Declaration in support of the responsive arguments until the undersigned received the Declaration by courier from Japan on October 15, 2003.

Accordingly, Applicants respectfully request that the attached declaration be joined with the response filed October 14, 2003.

Very respectfully,

Dated: 10/15/03


Dennis G. LaPointe
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Reg. No. 40,693

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Practitioner's Docket No. 1581.29

PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of: Shigeto Matsumura et al.

Application No.: 09/761,043

Group No.: 1712

Filed: 01/15/2001

Examiner: D.S. Metzmaier

For: COLLOIDAL SILICA SLURRY

Commissioner for Patents

P.O. Box 1450

Alexandria, VA 22313-1450

EXPRESS MAIL CERTIFICATE

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Marigelle Malapira, Legal Assistant

A handwritten signature in black ink, appearing to read "Marigelle Malapira".

Signature of person mailing paper or fee



1 UNITED STATES DEPARTMENT OF COMMERCE
2 PATENT AND TRADEMARK OFFICE

3
4 Applicant : Shigetoyo Matsumura)
5 Serial No. : 09/761,043) ART UNIT: 1712
6 Filing Date : January 15, 2001) EXAMINER:

7 Daniel S. Metzmaier

8 Title : Colloidal Silica Slurry

9 Commissioner of Patents and Trademarks

10 Washington, D.C. 20231

11 For: Colloidal Silica Slurry

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12
13 DECLARATION
14
15 I, Masatoshi Sakai, hereby declare as follows.

16
17 1. I was born on January 24, 1949, in Nanao city, Ishikawa prefecture,
18 Japan, and graduated from Yokohama National University, Faculty of
19 Engineering on March 1971. My major was safety engineering.

20
21 After graduating from Yokohama National University, I had worked
22 research and development of colloidal silica in Tonen Chemical Corporation
23 from 1985 to December 1998, and thereafter, I have worked research and
24 development of colloidal silica in Fuso Chemical Co., Ltd., since January
25 1999.

26
27 2. Here is a list of patent applications that I have invented.

28 Tokuganhei 4-180496 (No. 4-180496) "Epoxy oxygen composition for sealing

1 semiconductor material"

2 Tokuganhei 9-363239 (No. 9-363239) "Manufacturing method of silica glass

3 powder particle"

4 Tokuganhei 10-141064 (No. 10-141064) "Polish abrasive coating, abrasive

5 and method of polish"

6 Tokuganhei 10-202844 (No. 10-202844) "Silica glass powder particle and its

7 manufacturing method"

8 Tokugan 2000-236453 (No. 2000-236453) "Colloidal Silica Slurry"

9 Tokugan 2000-236454 (No. 2000-236454) "Colloidal Silica Slurry"

10 Tokugan 2001-360595 (No. 2001-360595) "Nonporous spherical silica and its

11 manufacturing method"

12 Tokugan 2001-360596 (No. 2001-360596) "Manufacturing method of

13 hydrophobic lightweight porous silica gel"

14 Tokugan 2002-67585 (No. 2002-67585) "Nonporous spherical silica and its

15 manufacturing method"

16 Tokugan 2002-251019 (No. 2002-251019) "Manufacturing method of high

17 purity hydrophilic organic solvent dispersion silica sol, high purity

18 hydrophilic organic solvent dispersion silica sol obtained from the

19 manufacturing method, manufacturing method of high purity organic

20 solvent dispersion silica sol and high purity organic solvent dispersion silica

21 sol obtained from the manufacturing method"

22

23 3. I am one of the inventors of US Serial No. 09/761,043 Colloidal Silica

24 Slurry, and have a full knowledge of the content thereof.

25

26 4. Claims of the present invention are described below.

27

28 9. The colloidal silica slurry wherein

1 it does not have a bad influence, such as corrosion, to a silicon wafer
2 and wiring material on a silicon wafer and inhibits growth of microbes, and
3 whereof preserving stability is high because stability of a particle diameters
4 of colloidal particle is superior and using for a long period continuously is
5 possible, and

6 said colloidal silica slurry is comprised by adding hydrogen peroxide
7 5 ~ 100 ppm to lowered metal silica slurry which metal content is 1 ppm or
8 less and produced from silicate ester, and the pH of colloidal silica slurry is
9 6.0 ~ 8.0 and content of colloidal silica is 0.05 ~ 50 weight %.

10
11 13. The colloidal silica slurry as claimed in claim 9, wherein pH is
12 from 6.5 ~ 7.5.

13
14 14. The colloidal silica slurry as claimed in claim 9, wherein
15 additional quantity of said hydrogen peroxide is from 10 ~ 50 ppm.

16
17 15. The colloidal silica slurry as claimed in claim 9, wherein average
18 particle diameter of said colloidal silica is from 5 ~ 300 nm.

19
20 16. The colloidal silica slurry as claimed in claim 9, wherein average
21 particle diameter of said colloidal silica is from 10 ~ 250 nm.

22
23 5. The above-mentioned patent was derived from the test examples as
24 below described in original specification.

25 (Test example 1; Decomposed test 1 of hydrogen peroxide in the colloidal
26 silica)

27 Samples whereof 100 ppm, 50 ppm, 30 ppm, 20 ppm, and 10 ppm of
28 hydrogen peroxide were added respectively to high purity colloidal silica

1 (Product name: Quartron PL-10, Made by Fuso Chemical Co., Ltd.) were
2 made. After they were preserved for one week at 37 °C, 5 ml of each was
3 extracted, 1 ml of vanadium sulfate was added to this, and remained amount
4 of hydrogen peroxide was measured by (visual) comparing to the coloring of
5 comparative samples prepared previously.

6 Furthermore, comparative samples were prepared by adding 1 ml of
7 vanadium sulfate to 5 ml of samples whereof 100 ppm, 50 ppm, 30 ppm, 20
8 ppm, and 10 ppm of hydrogen peroxide were added respectively to colloidal
9 silica.

10 This result is shown in table 1.

11

12 Table 1

13

Added concentration	Concentration of Hydrogen Peroxide (ppm)				
	100	50	30	20	10
Immediately after adding	100	50	30	20	10
One day later	100~70	50~40	30~20	20~10	7~5
One week later	100~70	50~40	30~20	20~10	7~5
Two weeks later	100~70	50~40	30~20	20~10	7~5
One month later	100~70	50~40	30~20	20~10	7~5

14

15

16 As the result in table 1, after the samples were preserved for one day
17 under the condition of 37 °C, the decomposition of hydrogen peroxide, even it
18 is a little, was observed. However, the decreasing of remained amount of
19 hydrogen peroxide, which was passed one week, two weeks, and one month,

1 was not observed. Therefore, the decomposition of hydrogen peroxide is
2 observed a little in a short period; however, there is no more decomposition in
3 a medium and long period so that the hydrogen peroxide is preferably used
4 as antibacterial biocide of the colloidal silica slurry.

5

6 (Test example 2; Inhibiting test 1 of bacteria growth by adding hydrogen
7 peroxide)

8 Samples, whereof 500 ppm, 100ppm, 50 ppm, and 10 ppm of
9 hydrogen peroxide were added respectively to 10 ml of high purity colloidal
10 silica (Product name: Quartron PL-10, Made by Fuso Chemical Co., Ltd.)
11 were made. 1 ml of bacteria (A number of bacteria: $5.1 \times 10^6 / \text{ml}$) which
12 was extracted from colloidal silica containing heat resistant sporocarp and
13 the like was added. Each 1 ml was extracted and was measured a number
14 of bacteria after it passed one week, two weeks, and one month.

15 Furthermore, each 1 ml of sterilized culture medium was added to
16 each sample passed one month, then after it was cultivate for two days at
17 37 °C, the existence of bacteria (final number of bacteria) in testing solution
18 was examined.

19
20
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28

This result is shown in table 2.

Table 2

(Unit: CFU / ml)

	Added concentration	hydrogen peroxide			
		500ppm	100ppm	50ppm	10ppm
Bacteria extracted from colloidal silica	One week later	0	0	0	0
	Two weeks later	0	0	0	0
	One month later	0	0	0	0
	Final number of bacteria	0	0	0	0

As the result in Table 2, no bacterium was detected even after one month cultivation. Additionally after adding culture medium finally, bacterium was not detected at all. Therefore, the effect of hydrogen peroxide as antibacterial biocide can be maintained at low concentration for a long period.

(Test example 3; Inhibiting test 2 of bacteria growth by adding hydrogen peroxide)

Bacteria extracted from colloidal silica was added to a sample whereof hydrogen peroxide or ammonia was added to sterilized water, then how a number of bacteria changes was measured by adding hydrogen peroxide or pH adjustment with adding ammonia.

First, samples were made with adding ammonia or hydrogen peroxide to 10 ml of sterilized water respectively to become: for the ammonia, each pH value in table 3; and for the hydrogen peroxide, each concentration in table 3. Next, cultivation solution of bacteria extracted from colloidal

silica was diluted to two stages (high concentration; 5.8×10^3 CFU / ml, low concentration; 58 CFU / ml), each 1 ml of the cultivation solution was added to each sample, and a number of bacteria passed one hour was measured with a filter filtration method. This result is shown in table 3.

Moreover each sample whereof 500 ppm, 100 ppm, 50 ppm, 10 ppm, and 5 ppm of hydrogen peroxide were added respectively to high purity colloidal silica (Product name: Quartron PL-10, Made by Fuso Chemical Co., Ltd.) was made, and each 1 ml of cultivation solution which is diluted to two stages (high concentration; 5.8×10^3 CFU / ml, low concentration; 58 CFU / ml) as same as the above is added to each sample. After it was passed one week, 1 ml of the culture medium was added, and the existence of growth of bacteria which passed two more days was measured. This result is shown in table 4.

Table 3

(Unit: CFU / 11ml)

Bacteria solution	Blank	Ammonia				Hydrogen peroxide					
		pH 9.5	pH 10.0	pH 10.5	pH 11.0	500 ppm	100 ppm	50 ppm	10 ppm	5 ppm	1 ppm
High concentration	200<	200<	200<	200<	200<	1	57	152	61	200<	116
Low concentration	40	57	49	51	49	0	0	0	0	5	2

1 Table 4
2

Bacteria solution	Blank	Hydrogen peroxide				
		500ppm	100ppm	50ppm	10ppm	5ppm
High concentration	Growing	Not grown	Not grown	Not grown	Not grown	Not grown
Low concentration	Growing	Not grown	Not grown	Not grown	Not grown	Growing

3
4 As the result in Table 3, even if the pH of sterilized water is prepared
5 by ammonia, growth of bacteria can not be inhibited. On the other hand,
6 when hydrogen peroxide is added, it is found that growth of bacteria can be
7 inhibited even with a little adding like 1 ppm.

8 Also, as the result in Table 4, when hydrogen peroxide is added to
9 colloidal silica, it is found that growth of bacteria can be inhibited with
10 adding approximately 10 ppm or more of hydrogen peroxide.

11
12 (Test example 4; Preserving test of colloidal silica adding antibacterial
13 biocide)

14 High purity colloidal silica (Product name: Quartron PL-10, Made by
15 Fuso Chemical Co., Ltd.) wherein 10 ppm and 100 ppm of hydrogen peroxide
16 were added respectively was preserved in sealed container under the
17 condition of 25 °C, and average of particle diameters and pH value, which
18 passed 24 hours from adding hydrogen peroxide and which passed six
19 months, were measured. The average of particle diameters was measured
20 with using Sub-Micron Particle Analyzer (Product Name: Coulter Model N4,
21 Made by Coulter Electronics, Inc.). Furthermore, high purity colloidal silica
22 wherein hydrogen peroxide was not added was measured as a blank. This

1 result is shown in table 5.

2

3 Table 5

4

	Added amount (ppm)	After 24 hours passed		After 6months passed	
		pH	Average of particle diameter (nm)	pH	Average of particle diameter (nm)
Hydrogen peroxide	10	7.1	220	6.9	221
Hydrogen peroxide	100	7.0	221	6.8	220
Blank	-	7.1	221	6.8	219

5

6 As the result in Table 5, it is found that colloidal silica wherein
7 hydrogen peroxide as antibacterial biocide is added, even it is preserved for a
8 long period, is superior on preserving stability because it hardly affects to the
9 average of particle diameters and pH value which are said that they are easy
10 to be affected most.

11

12 6. I considered four references (US 3,860,431, US 5,230,833,
13 JP61-209909 and US4,973,462) cited by the Examiner.

14 (1) In the US 5,230,833 (Romberger et al), lowered metal silica slurry
15 used in polishing of silicon wafer is described, and it is also described that
16 lowered metal silica can be produced from silicate ester.

17 In the column "BACK GROUND OF THE INVENTION", it is
18 described as "in the US Patent No. 3,860,431, hydrogen peroxide is used as
19 biocide".

1 In line 59 to 65, column 15, it is described as "Al, present in an
2 amount less than about 100ppm, based on SiO₂; iron, as Fe, present in an
3 amount less than about 50ppm, based on SiO₂; potassium, as K, present in an
4 amount less than about 25ppm, based on SiO₂; and sodium, as Na, present in
5 an amount less than about 500ppm, based on SiO₂".

6 In the US 3,860,431 (Payne et al), an invention which relates to slip
7 resistant composition for paper coating is described, and in line 50 ~ 58 of
8 fourth column, hydrogen peroxide is shown as a kind of a biocide.

9 In line 64 to 66, column 4, composition quantity of biocide is
10 described as 0 ~ 0.5% and preferably 0.3 ~ 0.5 %.

11 (2) In the US 5,230,833, it is described as "pH, as prepared with
12 ammonia, ranging from between about 8.5-11.3" (line 34 to 35, column 8),
13 and pH is clearly described as pH 8.5 ~ 11.3.

14 From this description, in the invention of the US 5,230,833, the
15 colloidal silica slurry cannot be set less than pH 8.5.

16 In the US 3,860,431, the colloidal silica slurry is described as pH 8.6
17 ~ 10.0, so the colloidal silica slurry cannot be set less than pH 8.6.

18 Further, in the invention of the US 3,860,431, even if one skilled in
19 the art selecting hydrogen peroxide as biocidal additive is not easy. In
20 alkaline colloidal silica slurry such as described in the US 3,860,431, even if
21 hydrogen peroxide is added, it resolves and does not work as biocidal
22 additive. By testing, one skilled in the art can easily understand that in the
23 invention of the US 3,860,431, in four biocidal additives, hydrogen peroxide
24 cannot be used in practice.

25 (3) Moreover, in the US 5,230,833, content of metal impurity is described.
26 Content of four types of metal impurities described in claim 1 is less than 675
27 ppm, but content of metal impurity of the present invention is prepared 1
28 ppm or less. There is a big difference in these contents. In the present

1 invention, content of metal impurity is prepared as 1 ppm or less, because
2 bad effect to electric properties of semiconductor parts and decomposition of
3 hydrogen peroxide composed as biocidal additive can be prevented. In the
4 US 3,860,431, there is no clear definition of content of metal impurity.

5 (4) In the present invention, decomposition of hydrogen peroxide can be
6 prevented for long period by preparing colloidal silica slurry pH 6.0 ~ 8.0 and
7 by preparing content of metal impurity 1 ppm or less, and this is described in
8 after-mentioned test example. In the test example, an evaluation of
9 decomposition of hydrogen peroxide by preparing of colloidal silica slurry pH
10 7.4 ~ 9.5 and by adding hydrogen peroxide into said colloidal silica slurry is
11 shown.

12 In the last declaration, a test example evaluating decomposition of
13 colloidal silica slurry with using commercially available colloidal silica slurry
14 which is pH 9.6 and 9.2 and by adding hydrogen peroxide into the colloidal
15 silica slurry is shown.

16 Even though the colloidal silica slurry has almost same pH, in a
17 sample (pH 9.5) of after-mentioned test example that content of metal
18 impurity is 1 ppm or less, substantive decreasing of hydrogen peroxide
19 decomposition is shown as compared to a sample (pH 9.6 or 9.2) which I
20 submitted last that has over 1 ppm of metal impurity content.

21 Further, in after-mentioned test example, as increasing pH,
22 increasing of decomposition of hydrogen peroxide is shown.

23 Therefore, to prevent decomposition of hydrogen peroxide in colloidal
24 silica slurry, pH is needed to be prepared 6.0 ~ 8.0 and content of metal
25 impurity is needed to be prepared 1 ppm or less.

26 (5) As mentioned above, in the invention of '833 which cites invention
27 and description of '431, hydrogen peroxide is shown as biocidal additive of
28 silica sol, but in practice, hydrogen peroxide cannot be used as biocidal

1 additive. In JP61-209909, a method for producing colloidal silica slurry
2 from silicate ester is described, but the colloidal silica slurry is prepared pH
3 9.0 ~ 10.0. The colloidal silica slurry is different to colloidal silica slurry of
4 the present invention, which is prepared pH 6.0 ~ 8.0. Further, in US
5 5,230,833, a method for producing colloidal silica slurry from silicate ester is
6 described, but preparing metal impurity content 1 ppm or less and preparing
7 pH 6.0 ~ 8.0 are not described, and the colloidal silica slurry is different to
8 the colloidal silica slurry of the present invention, which metal impurity
9 content is prepared 1 ppm or less and pH is prepared pH 6.0 ~ 8.0.

10 (6) On the other hand, the colloidal silica slurry which relates to the
11 present invention is comprised by adding hydrogen peroxide 5 ~ 100 ppm to
12 lowered metal silica slurry which metal content is 1 ppm or less and
13 produced from silicate ester. The pH of colloidal silica slurry is 6.0 ~ 8.0 and
14 content of colloidal silica is 0.05 ~ 50 weight %.

15 The colloidal slurry does not have a bad influence, such as corrosion,
16 to a silicon wafer and wiring material on a silicon wafer and inhibits growth
17 of microbes, and whereof preserving stability is high because stability of a
18 particle diameters of colloidal particle is superior and using for a long period
19 continuously is possible. Content of metal impurity of colloidal silica slurry
20 is prepared 1 ppm or less and pH is prepared 6.0 ~ 8.0. By preparing
21 content of metal impurity 1 ppm or less and pH 6.0 ~ 8.0, the colloidal slurry
22 can prevent decomposition of hydrogen peroxide and can inhibit growth of
23 microbes for a long period. Further, the colloidal slurry does not have a bad
24 influence, such as corrosion, to a silicon wafer and wiring material on a
25 silicon wafer.

26 When colloidal silica slurry described in such as the US 5,230,833
27 (Romberger et al) or the US 3,860,431 (Payne et al) is over pH 8.5 and when
28 content of metal impurity is over 1 ppm, hydrogen peroxide decomposes in a

1 short period, and cannot obtain effect that the present invention has.
2 Further, the colloidal silica slurry has a risk to have a bad influence, such as
3 corrosion, to a silicon wafer and wiring material on a silicon wafer.

4 (7) As explained above, in the inventions of Romberger et al and Payne
5 et al that preparing pH which is over 8.5 is described but preparing pH
6 which is less than 8.5 and preparing content of metal impurity which is 1
7 ppm or less is not described, even if one skilled in the art, it is not easy to
8 figure out preparing pH 6.0 ~ 8.0 and preparing metal impurity less than 1
9 ppm. Further, even if one skilled in the art, it is not easy to figure out to
10 obtain colloidal silica slurry which can prevent decomposition of hydrogen
11 peroxide and have high stability by preparing pH less than 8.0 and
12 preparing content of metal impurity 1 ppm or less. Therefore, the present
13 invention has inventive step.

14

15 7. To prove above-mentioned my theory, I describe a test example.

16

17 I Object of the test

18 To test decomposition of hydrogen peroxide in the colloidal silica
19 which was prepared neutral zone and alkaline zone.

20

21 II Sample

22 As sample, 100 ppm hydrogen peroxide which concentration is 30%
23 was added to extra high purity colloidal silica (Product name: PL-3, Made by
24 Fuso Chemical Co., Ltd.), and then, pH of the colloidal silica slurry was
25 prepared to 8.0, 8.5 and 9.5 by using ammonia water and potassium
26 hydroxide aqueous liquor.

27 Physicality value of the extra high purity colloidal silica is listed on
28 table 2. The physicality values are slightly different to physicality values of

1 colloidal silica slurry of test example in the last reasoning and declaration,
2 because a production lot is different.

3

4 III Test method

5 The samples were preserved at 40 °C and measured change of
6 concentration of hydrogen peroxide. A determination method of
7 concentration of hydrogen peroxide was followed iodometric titration method
8 which adds potassium iodide to each sample and titrates generating Iodine
9 with sodium thiosulfate solution.

10

11 IV Result

12

13 Test result of samples which were preserved under thermal condition
14 40 °C for 12 days is described in Table 6.

15

16 Table 6

17

(ppm wt/wt)

	p H	Before Test	40°C After 12 days
Fuso Chemical Co., Ltd. PL-3 +H ₂ O ₂	7.41	81.5	81.4
Add NH ₃	8.02	81.5	81.2
Add NH ₃	8.48	81.5	81.1
Add NH ₃	9.48	81.5	80.0
Add KOH	8.00	81.5	79.6
Add KOH	8.50	81.5	78.1
Add KOH	9.50	81.5	75.9

18

19

20

1 Table 7

Company	Fuso Chemical Co., Ltd.	
Product name	Quartron PL-3	
pH	7.5	
specific gravity (20/4°C)	1.120	
concentration of Silica wt%	19.50	
BET specific surface area m ² /g	77	
primary particle diameter (conversion method using specific surface area) nm	35.6	
metal impurity		
Na ppm	0.07	
K ppm	0.01 or less	
Fe ppm	0.005 or less	
Al ppm	0.01 or less	
Ca ppm	0.01 or less	
Mg ppm	0.01 or less	
Ti ppm	0.5 or less	
Ni ppm	0.005 or less	
Cr ppm	0.005 or less	
Cu ppm	0.005 or less	

3
4 As mentioned in Table 6, decomposition of hydrogen peroxide is
5 recognized as increasing pH values, and compared to a sample which content
6 of metal impurity is over 1 ppm, a degree of decomposition of hydrogen
7 peroxide decreases drastically.

8 Therefore, to use hydrogen peroxide as biocidal additive of colloidal

1 silica slurry, pH is needed to be prepared 8.0 or less and content of metal
2 impurity is needed to be prepared 1 ppm or less.

3

4 8. Conclusion

5 As explained above, the present invention and inventions of cited
6 references are different. Further, the present invention has superior effects
7 that can inhibit decomposition of hydrogen peroxide in the colloidal silica
8 slurry and obtain high preserving stability, and these effects cannot be
9 figured out from each cited reference. Therefore, the present invention has
10 an inventive step.

11

12 , October, 10,2003

13

14 Masatoshi Sakai

15

16 Masatoshi Sakai